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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 7 DETERMINATION OF ANTIMONY BY RHODAMINE B SPECTROPHOTOMETRIC METHOD

(First Revision)

UDC 669·35'6 : 543·24 [546·86]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee, MTD 28

FOREWORD

This Indian Standard (Part 7) (First Revision) was adopted by the Bureau of Indian Standards on 25 May 1990, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This Indian Standard was first published in 1967 as Methods of chemical analysis of bronzes and covered determination of copper, lead, zinc, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing, the Sectional Committee responsible for the preparation of this standard felt that it shall be convenient to bring it out in a series of parts which, on publication will supersede the relevant methods for determination given in IS 4027 : 1967. This (Part 7) is one of that series and covers the determination of antimony by Rhodamine B spectrophotometric method. The other parts are as follows:

Part 1 Determination of copper and lead by electrolytic method
Part 2 Determination of manganese by photometric method
Part 3 Determination of phosphorus by volumetric method
Part 4 Determination of nickel by photometric method
Part 5 Determination of tin by iodimetric method
Part 6 Determination of zinc by complexometric (EDTA) method

Methods for chemical analysis of other constituents in bronzes are under preparation and will be published as subsequent parts of above series.

In the present revision determination of antimony by the potassium bromate (volumetric) method has been replaced by Rhodamine B spectrophotometric method and brought up in line with ISO 5956 : 1984 (E) 'Copper and Copper alloys — Determination of antimony content — Rhodamine B spectrometric method issued by the International Organization for Standardization.

The method of analysis prescribed in this standard may be used by the laboratories for their day to day work. It shall also be a referee method in cases of dispute.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 7 DETERMINATION OF ANTIMONY BY RHODAMINE B SPECTROPHOTOMETRIC METHOD

(First Revision)

1 SCOPE

1.1 This standard covers method for determination of antimony in the range from 0·02 to 0·05 percent in bronzes.

2 REFERENCE

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)
1817 : 1961	Methods of sampling of non-ferrous metals for chemical analysis

3 SAMPLING

3.1 Sample shall be drawn and prepared in accordance with IS 1817 : 1961.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF ANTIMONY BY RHODAMINE B SPECTROPHOTOMETRIC METHOD

5.1 Outline of the Method

Antimony is extracted with *iso*propyl ether from the chloride solution and treated with rhodamine B. Absorbance of the red colour complex of chloroantimonate Rhodamine B formed is measured at 550 nm.

5.2 Reagents

5.2.1 *Concentrated Hydrochloric Acid,* rd 1·16 (conforming to IS 265 : 1987).

5.2.2 *Hydrogen Peroxide,* 30 percent (*v/v*).

5.2.3 *Dilute Hydrochloric Acid,* 1 : 2 (*v/v*).

5.2.4 *Cerium (IV) Sulphate Solution*

Dissolve 4 g of cerium sulphate Ce (SO_4) 2·4 H_2O and dilute to 100 ml with dilute sulphuric acid (1 percent).

5.3.5 *Hydroxylammonium Chloride,* 1 percent (*w/v*)

5.3.6 *Isopropyl Ether*

5.3.7 *Rhodamine B Solution*

Dissolve 0·01 g of Rhodamine B and dilute to 100 ml with dilute hydrochloric acid (10 percent).

5.3.8 *Standard antimony Solution — (1 ml = 0·5 mg Sb)*

Dissolve 100 mg of antimony metal (purity 99·9 percent, *Min*) in 20 ml concentrated sulphuric acid. Fume for 5 minutes and cool. Dilute to 150 ml with dilute sulphuric acid (1 : 9). Transfer to 200 ml volumetric flask. Cool and dilute to volume with dilute sulphuric acid (1 : 9) and mix.

5.3.8.1 *Standard antimony solution (1 ml =* 2·5 *µg Sb)*

Transfer 5 ml of antimony solution (1 ml = 0·5 mg Sb) to a one litre volumetric flask containing 200 ml of dilute sulphuric acid (50 percent). Dilute to volume with distilled water and mix thoroughly.

5.4 Procedure

5.4.1 Dissolve 0·5000 g of sample in 15 ml concentrated hydrochloric acid. Add dropwise 5 to 10 ml of hydrogen peroxide. Cool until the violent reaction ceases. Heat the solution for 2 to 3 minutes. Cool, transfer to 200 ml volumetric flask and make the volume with dilute hydrochloric acid. Transfer 1 ml of aliquot (containing about 10 µg of antimony) and 15 ml of concentrated hydrochloric acid (cooled to 5°C) to a separating funnel.

5.4.2 Add 2 ml of cerium sulphate and mix. After 2 minutes add 0·5 ml of hydroxylammonium chloride solution and mix. Add 10 ml of *iso*propyl ether (cooled to 5°C) and shake for 15 seconds. Allow to stand for 5 minutes and discard the aqueous layer. Wash the organic layer twice with 5 ml dilute hydrochloric acid (1 : 10). Add 20 ml of Rhodamine B solution and shake for 30 seconds and allow to stand for 2 minutes. Drain the aqueous layer together with small portion of organic layer. Collect the remaining organic layer in a dry, stoppered flask. Measure the absorbance against water at 550 nm.

5.4.3 *Calibration Curve*

Transfer 0·500 g antimony free copper into each of seven conical flasks. Add 0, 1, 2, 3, 4, 5 and 6 ml of standard antimony solution (1 ml = 2·5 µg Sb) and proceed as in **5.4.1** and **5.4.2** using same quantity of all the reagents. The solutions for spectrophotometric measurements thus prepared will contain 0 to 15 µg of Sb. Measure the absorbance values of each solution against water. Plot a calibration curve of absorbance values against micrograms of antimony per ml of solution.

5.4.4 *Calculation*

Convert the spectrophotometric readings of test solution to/µg of antimony by means of calibration curve. Calculate the percentage of antimony as follows:

$$\text{Antimony, percent by mass} = \frac{A \times 100}{B \times 10^6}$$

where

A = mass, in µg of antimony found in aliquot of sample taken; and

B = mass, in g, of sample represented in the aliquot taken for the test.

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